PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5:

(11) International Publication Number:

WO 90/04960

A61K 7/46

A1

(43) International Publication Date:

17 May 1990 (17.05.90)

(21) International Application Number:

PCT/US89/04958

(22) International Filing Date:

3 November 1989 (03.11.89)

(30) Priority data:

266,792 428,176 3 November 1988 (03.11.88) US US 27 October 1989 (27.10.89)

(60) Parent Application or Grant (63) Related by Continuation US

266,792 (CIP)

Filed on

3 November 1988 (03.11.88)

(71)(72) Applicant and Inventor: KING, Michael, L. [US/US]; 1410 St. Louis Bridge Road, Decatur, IL 62521 (US).

(74) Agent: FRENCH, Timothy, A.; Fish & Richardson, One Financial Center, Suite 2500, Boston, MA 02111-2658

(81) Designated States: AT, AU, BB, BE (European patent), BF (OAPI patent), BG, BJ (OAPI patent), BR, CF (OAPI patent), CG (OAPI patent), CH, CM (OAPI patent), DE, DK, FI, FR (European patent), GA (OAPI patent), GB, HU, IT (European patent), JP, KP, KR, LK, LU, MC, MG, ML (OAPI patent), MR (OAPI patent), MW, NL, NO, RO, SD, SE, SN (OAPI patent), SU, TD (OAPI patent), TG (OAPI patent), US.

Published

With international search report.

Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(54) Title: FRAGRANT MATERIAL

(57) Abstract

A fragant material that imparts a pleasant fragrance when set out at ambient temperature or when placed in heated water includes an aggregate of small sodium chloride granules such as are produced by vacuum granulation, the granules formed into a molded shape, e.g. as a plurality of discrete pellets, or in a loose state, and having a fragment oil and optionally a water soluble dye dispersed in the material. Also disclosed is a fragrant material consisting of the combination of salt granules with a minor amount of finely divided inert particles, and a water-soluble dye. Other features that enhance performance and enable the product to be of low cost are also described.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	ES	Spain	MG	Madagascar
ΑU	Australia	FI	Finland	ML	Mali
BB	Barbados	FR	France	MR	Mauritania
BE	Belgium	GA	Gabon	MW	Malawi
BF	Burkina Fasso	GB	United Kingdom	NL	Netherlands
BG	Bulgaria	HU	Hungary	NO	Norway
		ır	Italy	RO	Romania
BJ	Benin	JP	Japan	SD	Sudan
BR	Brazil	KP	Democratic People's Republic	SE	Sweden
CA	Canada	N.F	of Korea	SN	Senegal
CF	Central African Republic	***	+	SU	Soviet Union
CG	Congo	KR	Republic of Korea		
CH	Switzerland	Ц	Liechtenstein	TD	Chad
CM	Cameroon	LK	Sri Lanka	TG	Togo
DE	Germany, Federal Republic of	w	Luxembourg	us	United States of America
שב	Denmark	MC	Manaco		

- 1 -

FRAGRANT MATERIAL .

Background of the Invention

This invention relates to fragrant material for imparting a pleasant fragrance to a room, and the like.

5 Man has imparted pleasant fragrances to rooms since time immemorial. Flowers, eucalyptus leaves, dill, and incense have been used for this purpose for thousands of years and are still widely used today. A wide variety of fragrant oils are now commercially available, but they are rarely used by themselves for providing fragrance to rooms 10 because of their rapid evaporation rate and because of the danger of spills. Instead, fragrant oils are generally incorporated into a solid carrier, also known as a fixative. A number of different prior art carriers are or have been used with fragrant oils. For example, Moore U.S. 4,427,366 15 discloses wax chips containing a fragrant oil and Gyulay U.S. 4,579,717 discloses a porous ceramic ring which absorbs a fragrant oil. Another well known carrier is a mixture of dried Orris root, dried Calamus root, and other dried plant material which, when scented with a fragrant oil, is 20 commonly sold as potpourri. Rock salt to which fragrance oils have been applied has also been sold. However, none of these carriers is ideal.

An ideal carrier for a fragrant oil, depending upon
the application, would exhibit many specific properties.
First, the carrier would readily absorb fragrant oils and
would leave no surface film which might cause staining or
tackiness. Second, the carrier would also readily absorb
dyes to permit the carrier to be dyed a color corresponding
to the fragrance if desired. Third, the carrier would be
odorless so as to not clash with the fragrance of the oil.
Fourth, it would be nonflammable, nontoxic, nonallergenic,
non-nutritive and environmentally safe for obvious safety

- 2 -

reasons. Fifth, for particular applications, an ideal carrier would consist of discrete particles having an average size of about 1/4 to 1 inch (6.35 to 25.4 mm) with little or no fines. Such particles can be easily mixed, poured, and handled; they attractively fill up bowls and containers of various sizes and shapes; and, if spilled, can be picked up by hand. Sixth, the carrier would not soften or melt at elevated temperatures of 100 to 130°F (38 to 55°C) which are encountered in closed houses and automobiles during summer months. Seventh, the carrier would be relatively inexpensive and readily available.

Furthermore, an ideal carrier would be suitable for "simmering" - warming the material in or out of water to accelerate the flow of fragrance into the room. More particularly, the carrier would dissolve slowly in water, cause no harm to pots and pans and permit an easy clean-up after use.

10

15

20

25

30

A large number of potential carriers were considered and/or tested and then rejected before the fragrant material of this invention was discovered. For example, potpourri crumbles into fines and leaves a messy residue aftersimmering in hot water. Wood chips exhibit similar disadvantages and have too strong an odor. Sugar cubes readily absorb oils and dyes, but become sticky and attract bugs. Starch readily absorbs oils and dyes, but cannot be easily formed into lasting solid shapes, attracts bugs and leaves a messy residue when used with water. Rock salt does not absorb oil and dye well because of its structure. Wax chips must be melted to incorporate the fragrant oil and dye, release little fragrance until they are heated, and create a mess if placed directly into hot water. ceramic materials have limited absorbency, are expensive, and present a danger if broken because of the sharp and

- 3 -

jagged edges of the pieces. Cellulose fibers are dry and absorb oils and dyes, but are not water soluble and are expensive. Vermiculite is also dry and will absorb oils, but will not dye well, is not water soluble, and is expensive. Accordingly, until the fragrant material of this invention was discovered, there existed a strong demand for a better solid carrier for fragrant oils.

Summary of the Invention

Objects of this invention include to provide an improved fragrant material for imparting a pleasant fragrance to a room or the like. A more particular object is to provide such a fragrant material which can be set out at ambient temperature in a room or placed in heated water.

10

I have discovered a fragrant material which meets these objects. According to one aspect of the invention, 15 the material comprises a plurality of discrete pellets of compressed salt having a fragrant oil dispersed within the pellets. This fragrant material exhibits many advantages over other types of fragrant material which have been previously used in homes. The material has no surface film 20 so it is non-staining. The material is odorless, nonflammable, nontoxic, nonallergenic, non-nutritive, environmentally safe, homogeneous, non-crumbling with a high pressure break strength, and does not melt even at elevated temperatures. The material is relatively inexpensive and 25 can be easily mixed with other fragrant materials. Furthermore, this material dissolves slowly in water, does not harm pots and pans, and can be easily cleaned after use. Finally, because the fragrant oil, and dye if present, is uniformly dispersed throughout the pellets, a partial 30 dissolution of the pellets does not remove all the fragrant oil and dye from the pellets.

- 4 -

In another aspect, the invention features a fragrant material for imparting a pleasant fragrance when exposed in dry state at ambient conditions or when placed in heated water, that includes a molded shape of compressed sodium chloride salt granules having a fragrant oil dispersed within the molded shape.

Preferred embodiments of this aspect of the invention include additional features. A plurality of the molded shapes in the form of pellets are provided having an average size of approximately 1/4 to 1 inch (6.35 to 25.4 mm). The granules of the molded shape comprise vacuum granulated sodium chloride. The fragrant material additionally includes a dye dispersed within the molded The fragrant material is formed by the process of applying the fragrant oil to the surface of a pre-formed shape of the compressed salt granules and allowing the oil to be absorbed into the pores between the granules; preferably the oil is applied by spraying. The fragrant material is formed by the process of applying the fragrant oil to a loose aggregation of granules of sodium chloride and thereafter compressing the granules into the molded shape; preferably high molding pressure is employed to form the molded shape.

10

15

20

In another aspect, the invention features a fragrant
material for imparting a pleasant fragrance when exposed in
dry state at ambient conditions or when placed in heated
water, that includes an aggregation of small vacuum
granulated sodium chloride granules and a fragrant oil
dispersed within the aggregation. In addition to including
embodiments in which the aggregation of granules has been
formed into a molded shape, this aspect of the invention
also includes embodiments in which the small salt granules
remain loose as a powder.

In embodiments of the various aspects of the invention described above, certain additional features are preferred. Most of the sodium chloride granules are about 850 microns or less in size. The aggregation includes a minor amount of finely divided, inert, water insoluble, hygroscopic particles that increase the carrying capacity of the mixture for the fragrant oil; preferably these particles are selected from the group consisting of silicon dioxide, calcium silicate, magnesium carbonate, tricalcium phosphate 10 and sodium silicoaluminate and mixtures and combinations thereof; preferably they have a particle size of about 10 microns; preferably the weight ratio of the finely divided particles to the fragrant oil is about 1 to 3; preferably the finely divided particles comprise about 2% or more by weight of the fragrant material and more preferably to 15 enhance a fragrancing effect as described while still being economical, the finely divided inert particles comprise more than about 3% but less than about 10% by weight of the fragrant material. The fragrant oil of the fragrant material includes an organic carrier fluid selected from 20 diethylphthalate, methyl dihydroabietate, Hercolyn D, propylene glycol, dipropylene glycol, isoparaffins, Lozal, saltrol, polysorbate, D-limonene, glycerine and Isobar M; preferably the fragrant oil has a viscosity under ambient conditions of about 10 centipoise or more, preferably on the 25 order of between about 10 and 20 centipoise. The fragrant material further includes a dye which is soluble in water, and preferably also in oil; the dye preferably is a polymerbound azo colorant.

In another aspect, the invention features a dry, loose mixture comprising at least an aggregation of a large number of small vacuum granulated sodium chloride granules, a relatively minor amount of finely divided, inert, water

- 6 -

insoluble, hygroscopic particles and a fragrant oil dispersed within the aggregation.

5

10

15

20

25

30

Another aspect of the invention is a fragrant material for imparting a pleasant fragrance when exposed in dry state at ambient conditions or when placed in heated water, the fragrant material comprising an aggregation of small anhydrous salt granules and a minor amount of finely divided, inert, water soluble, hygroscopic particles, the finely divided inert particles selected from the group consisting of silicon dioxide, calcium silicate, magnesium carbonate, tricalcium phosphate and sodium silicoaluminate and mixtures and combinations thereof, the aggregation carrying a fragrant oil, the weight ratio of the finely divided particles to the fragrant oil being about 1 to 3, the salt granules in the fragrant material being selected so that in the presence of the inert particle they are dimensionally stable under ambient conditions.

Preferred embodiments also include selection of the ingredients to preform various functions. The fragrant oil is selected to perform as a room freshener. The fragrant oil is selected to mask a pervading odor such as, for example, an animal odor. The fragrant material is adhered as a coating to an object to which it is desired to impart a fragrance. A dry mixture may be, for example, formulated to mask a pervading odor, e.g., in the form of a rug freshener.

The invention also includes the methods of forming the various materials described above.

These and other features of the invention will be apparent from the following description of a presently preferred embodiment, and from the claims.

Description of a Presently Preferred Embodiment

In certain important embodiments of the invention
the fragrant material of this invention comprises discrete

pellets of compressed salt, fragrant oil, and, optionally, dye. If the fragrant material is going to be simmered in water, it is preferred that the salt contain minimum amounts of insolubles. If the fragrant material is not going to be simmered, the purity of the salt is not critical. 5 preferred type of salt is sodium chloride (NaCl) because of its low cost and ready availability. The physical form of the pellet is compressed granular salt. This form has a high pressure break strength and yet is still extremely 10 absorbent because of the microscopic pores between the compressed salt granules. For certain applications, the salt pellets have an average size of approximately 1/4 to 1 inch (6.35 to 25.4 mm), or about the size of marbles. many applications, pellets of a smaller size tend to 15 dissolve too quickly in water and are difficult to pick up if spilled, while pellets of a larger size dissolve too slowly in water and are not as attractive when placed in small bowls and containers. The shape of the pellet is not critical and spheres, cubes, cylinders, and the like are 20 acceptable. There is no requirement that the shape and size of the pellets be uniform and, in fact, some variation is aesthetically pleasing. One preferred form of salt pellet is of the type used commercially to recharge water softeners. A representative example of such a pellet is the Morton® System Saver™ water softening pellet. These pellets 25 typically contain about 99.70 percent sodium chloride and are about 1 inch (2.54 cm) long, 5/8 inch (1.59 cm) wide, and 3/8 inch (0.96 cm) thick.

A fragrant oil is a concentrated liquid fragrance.

The fragrant oil may be derived from natural sources or may be synthetic. The oil generally contains a variety of organic compounds of differing volatility. The particular type of fragrant oil is not critical to this invention and

- 8 -

many suitable fragrant oils are commercially available. A more complete discussion of fragrant oils is found at Volume 14, page 717, et seq. under the heading "Perfumes" in <u>Kirk-Othmer Encyclopedia of Chemical Technology</u> (2d Ed. 1967).

5

10

15

20

25

30

The optional dye is a substance which imparts color to the fragrant material. The dye may be available as a powder or as a liquid. Dyes which are water soluble are generally preferred because they have less tendency to stain and can be cleaned more easily. The particular dye used in this invention is a matter of choice and many suitable dyes having good color stability and retention are commercially available.

The relative amounts of salt pellets and fragrant oil in the material is a matter of choice which depends upon the strength of the fragrance in the oil and the desired strength of the fragrance in the fragrant material. The weight ratio of salt pellets to fragrant oil is generally about 20:1 to 100:1, and preferably about 30:1 to 50:1. If excessive amounts of fragrant oil are used, the saturation point of the salt pellets may be exceeded and the pellets may have an undesirable "wet" surface. If a dye is used, its quantity is also a matter of choice.

It is found while achieving a dry-to-the-touch character it is possible to include larger amounts of fragrant oil in the material by addition of the finely divided inert additive mentioned above in quantities greater than what might ordinarily be included as free-flowing agents in common salt. (In the prior art such agents have been used for the purpose of making salt free flowing or capable of being compressed into the form of a pellet.) The quantity of the particles is generally about 2% or more; in many cases, especially for free flowing powder embodiments,

- 9 -

in the range of about 3 to 10% of the total weight of the fragrant material is employed.

Further details concerning preferred ingredients and uses will be given following the description of certain examples.

5

10

15

20

Example 1

An 80 pound (36.3 kg) plastic film bag of Morton® Salt Pellets was opened and the contents gently poured into an enamel cast-iron 60 gallon (227 l) tub. The salt pellets are a commercial product of the Morton Salt Division of Morton Thiokol, Inc., Chicago, Illinois. They are compressed from vacuum granulated salt and contain a minimum of 99.5-percent sodium chloride. The dimensions of a typical pellet are 1 inch (2.54 cm) long, 5/8 inch wide (1.59 cm), and 3.8 inch (0.96 cm) thick.

Two pounds (0.98 kg) of peach fragrant oil, a commercial product of The Lebermuth Company, South Bend, Indiana, are poured into a standard 2.5 gallon (9.46 l) hand sprayer. Two ounces (57 gm) of Liquidtint Yello BL dye, a commercial product of the Millikin Dye Company, Inman, South Carolina, are then poured into the sprayer. The sprayer is then sealed and vigorously shaken to uniformly mix the fragrant oil and dye.

The sprayer is then pumped to generate pressure and
the contents are sprayed upon the pellets. The pellets are
mixed during spraying to obtain uniform distribution. After
spraying, the pellets are allowed to sit for about one hour
to allow complete absorption of the oil and dye. The
fragrant material is then screened to remove fines and
packaged.

Example 2

This example is formed by application of a fragrant oil to salt granules prior to compression of the granules to

- 10 -

form a pellet. A vanilla-creme fragrance containing vanilla-creme fragrant oil, Ethyl Vanillin, and Coumarin is obtained. (The fragrance supplier may include small quantities of other ingredients according to its recipe). The fragrance components are added to approximately 30% carrier fluid (Diethyl Phthalate) forming a fragrant oil with the following properties:

Specific Gravity @20°C : 1.1328-1.1338

(water =1)

10 Refractive Index @25°C : 1.5155-1.5165

Vapor Density (AIR=1) : >1

Boiling Point (of most

15 Appearance and odor : colorless to pale

yellow liquid

with a strong vanilla

fragrance.

The fragrant oil is then added to an anhydrous dendritic sodium chloride salt including about 2 percent of 20 Cabosil. About 2.5% of fragrant oil by weight is added to the salt (about 50 lbs. (22.7 kg) oil per 2000 lbs. (907.2 kg) of salt). The fragrant oil is mixed with the salt and Cabosil mixture to form a slurry. The slurry is gravity fed into a tablet press that exerts close to 1500 lb./sq. in. 25 (about 102 atms.) of pressure, achieving temperatures close to 806°C (near the melting point of NaCl granules at the high pressure), which results in fusion of the granules, with the formation of "bridging" areas where the granules are bonded together. After tabletizing the final pellets are extremely hard, shiny, solid and have a glazed appearance and are stable, non-crumbling, and smell like vanilla.

10

In use, with respect to the foregoing examples, there is no need of inducing an airflow because the fragrance is sufficiently strong to disperse on its own. The pellet or tablets will "fragrance" an average sized open room of 100 sq. ft. (9.3 ca) for at least 6 months, and are also highly water soluble.

A by-product of producing the pellets or tablets is the extra powder or granules that are vibrated off through a screen. This powder has all the properties of the pellets or tablets except that it is in powder form. It is found to have a number of practical applications, for instance, for placing in heated water, or as a fragrance powder, e.g. to mask a pervading odor such as the smell of urine.

Example 3

A fragrant material is formed by application of a fragrant oil to salt granules prior to compression of the granules to form a pellet. A cinnamon fragrance containing cinnamon fragrant oil, Cinnamine Aldehyde, Cinnamon Leaf Oil and Ethyl Vanillin is obtained. The fragrance components are added to approximately 20% carrier fluid (7% Hercolyn-D, 13% dipropylene glycol), forming a fragrant oil with the following properties:

Specific Gravity @20°C : 1.048-1.058

(water =1)

25 Refractive Index @25°C : 1.581-1.591

Vapor Density (AIR=1) : 1

Boiling Point (of most

volatile component 1%) : over 175°C
Solubility in water : Negligible

30 Appearance and odor : pale yellow liquid a

cinnamon fragrance.

The fragrant oil is mixed with dye. By volume, 3 cc yellow Chromatech dye and 9 cc red Chromatech dye per 1 lb.

- 12 -

(0.45 kg) of oil are used. The dyed fragrant oil is added to dendritic sodium chloride salt and about 2 percent Cabosil. About 2.5% of fragrant oil by weight is added to the salt (50 lb. oil (22.7 kg) per 2000 lb. (907.2 kg) of salt). The oil is mixed with the salt to form a slurry and processed as in Example 2.

Example 4

In this example, a loose form (powder) fragrant material is formed by application of a fragrant oil to a mixture of salt granules and particles that enhance carrying capacity. The material is formed as powder by addition of a fragrant oil to a non-compressed mixture of salt granules and a small amount of the finely divided particles. The amount of the enhancing particles is used in excess of 3% and generally less than about 10% by weight of the amount of salt, in this example, 5%.

A wintergreen fragrance containing wintergreen fragrant oil, peppermint, spearmint and wintergreen oil is obtained. The fragrance components are added to approximately 40% carrier fluid (isoparaffins), forming a fragrant oil with the following properties:

Specific	Gravity	020°C	:	0.885
•			•	0.005

(water =1)

5

10

15

20

25

30

Refractive Index @25°C : 1.464
Vapor Density (AIR=1) : >1

Boiling Point (of most

volatile component 1%) : 155°C

Solubility in water : Negligible

Appearance and odor : colorless to slightly

yellow liquid with a minty wintergreen

scent.

10

15

20

25

30

ij

The fragrant oil is mixed with dye. By volume, 1 cc yellow Chromotech dye and 0.1 cc blue Chromotech dye per 1 lb. (0.45 kg) of oil are used. The dyed fragrant oil is added to TFC 999 salt (salt treated at manufacture with yellow prussiate of soda anticaking agent) and about 5% calcium silicate. About 9.5% of fragrant oil by weight is added to the salt (95 lb. (43.1 kg) oil to 1000 lb. (453.6 kg) of salt). The oil is slowly added and mixed thoroughly with the salt and particles to form a loose dry mixture. The resulting product is a dry to the touch, free flowing and made up of hard, shiny small granules.

Due to the electrostatic properties of the salt the resulting powder may be easily applied in a spraying fashion to many different surfaces electrostatically. It also may be added to water for fragrancing a room by simmering.

Ingredients

Preferably, anhydrous vacuum granulated sodium chloride salt crystals are employed such as Morton Thiokol Food Grade 999 salt which has a granule size distribution of 98% less than 850 microns, and 51% less than 425 microns, (Morton Thiokol, Chicago, Ill.) or a vacuum granulated dendritic sodium chloride salt, e.g. Morton Thiokol Star Flake® or Dendritic ES® salt (latter includes 0.5% sodium silicoaluminate) which have a granule size distribution of 98% less than 425 microns and 65% less than 210 microns. Dendritic salts have a modified, high surface area, porous, crystalline structure which may enhance absorption of fragrant oil and therefore increase the life of the fragrant product or the amount of scent provided to the atmosphere or both. It will be understood that the granules may be much smaller, e.g. the size of a fine powder. In some cases, other anhydrous metal halide salts such as calcium or potassium chloride may be employed.

Examples of inert particles for enhancing the fragrant oil carrying capacity are particles which are inert, water insoluble, hygroscopic, ultrafine (generally less than 10 microns in size) particles such as silicon dioxide (e.g. Cabosil, Cabot Corporation available in some salts purchased from Morton Thiokol), calcium silicate, magnesium carbonate, tricalcium phosphate and sodium silicoaluminate (e.g. Zeolex available from J.H. Huber, Havre DeGrace, Maryland) or a combination thereof. Preferably, the particles are used when the amount of 10 fragrant oil required in the material is above the saturation point, i.e., the point where the amount of fragrant oil causes the material to become wet to the touch. For sodium chloride granules of the size here involved, the saturation point is about 2% by weight of fragrant oil. 15 finely divided particles are added at about one part (by weight) agent to every 3 parts fragrant oil. In pellet materials, the amount of fine particle agent is typically about 2% or more by weight. For example, to incorporate about 6.5% by weight of fragrant oil, the material may 20 include about 2% by weight of the enhancing particles. fragrant materials of loose granular form, the amount of the finely divided inert particles is typically greater than about 3% by weight, but in the interest of limiting cost, less than about 10%. It will be understood that 25 combinations of various types of particles can be formulated to take advantage of specific properties of the specific particles. For example, loose form fragrant materials may use a combination of silicon dioxide and magnesium carbonate. Silicon dioxide has greater fragrant oil holding 30 effectiveness, while magnesium carbonate is less dusty. will also be understood that an amount of particles greater than the 1 to 3 ratio may be used to enhance the free

flowing character of a loose mixture material or make pelletizing easier when applying fragrant oil to granules prior to pelletizing.

As previously indicated, it is well known that a fragrant oil is a concentrated liquid fragrance containing a 5 variety of organic compounds of differing volatility and viscosities. It is composed of natural and/or essential oils, and/or aroma chemicals (a liquid or crystalline fragrant chemical) and normally includes a carrier fluid 10 such as propylene glycol. The carrier fluids generally are non-fragrancing, non-volatile, miscible with the fragrant ingredients and non-reactive. Such fragrant oils are moderately viscous. A cinnamon fragrant oil as described in Example 3 has a viscosity of 10 cps at 25°C and vanilla 15 fragrant oil as described in Example 2, has a viscosity of 20 cps at 25°C (as measured on a Brookfield Viscometer model LVT at 30 rpm). A particular carrier fluid may be selected to enhance the physical properties such as dryness, hardness, and shiny appearance of the resultant fragrant 20 material. It may also be selected to change the evaporative rate of the fragrant oil thus to change the perceived strength in the sense of smell and the length of time of fragrancing. Examples of suitable carriers include diethylphthalate, Hercolyn (methyl dihydroabietate), Hercolyn hydrogenated methyl ester of Rosin (available as 25 Hercolyn D from the Lebermuth Co., Inc), propylene glycol, dipropylene glycol, isoparaffins, saltrol, Lozal (Co-C,, isoparaffins) (available from Shaw Mudge and Co.), Isobar M (isoparaffin) (available from R.N. Eaton and Co., Inc) polysorbate, D-limonene, and glycerin. The carrier fluid 30 can also be selected to serve as an anti-hardening agent in

powder form products. A combination of various carrier fluids can also be used.

5

20

25

30

Generally, an essential oil and additional carrier fluid are formulated prior to application to the granules to have a moderately high viscosity, i.e., preferably in the range of about 10 to 20 cps. If the viscosity is too low the fragrant oil will not remain on the salt and the material will be wet-to-the-touch. If too high, fragrancing will be insufficient.

10 For embodiments which apply fragrant oil prior to pelletizing, choice of press pressure can also affect the fragrancing strength. Increasing the press pressures, e.g. from 1500 lb/sq. in. to the range of 1600 lb/sq. in. to 1800 lb/sq. in. (about 110 to 125 atms.) allows the amount of fragrant oil to be decreased without sacrificing fragrant strength.

The dye, preferably water as well as oil miscible, may be added directly to the fragrant oil for application to the salt granules. The dye is typically a polymer bound azo colorant (e.g. Liquitints made by Milliken Chemical, Division of Milliken and Co. available from Chromatech, Inc.).

For producing a practical fragrant material with a new fragrance, one may follow the following procedures to achieve a strongly fragrancing product that lasts for a protracted duration and is dry-to-the-touch.

As a first step, a fragrance is obtained from a fragrance supplier. The combinations of chemical components for a particular fragrance that give rise to certain characteristic smells, also known as "notes", are selected, on a subjective trial basis to provide a pleasing odor, in general, as strong as possible. The fragrance may be selected by simply smelling a sample of the liquid. It will

be understood that the fragrant oil with a desireable scent may in some cases be simply purchased from a vendor and used directly as described herein, or sometimes it may be modified. For example, for a vanilla fragrance, a certain amount of coumarin may also be added to make it smell somewhat like cotton candy.

After selection of the fragrance, the fragrant oil is evaluated for use on salt granules. It is practical to begin with a fragrant oil that incorporates the carrier fluid ordinarily used by the fragrance vendor for such type of a fragrance.

10

15

The evaluation can use water softener pellets formed of salt granules (as in Example 1) regardless of whether the fragrant oil will be applied to a preformed pellet (as in Example 1), salt granules compressed into pellets after application of the fragrant oil (as in Examples 2 and 3) or salt granules that are to remain loose as a powder material (as in Example 4).

For the evaluation, a bag full of water softener pellets is thoroughly mixed with a two to five percent by 20 weight fragrant oil by pouring the oil in the bag, shaking the bag so the oil is coated around the pellets and taking 10 to 15 of the pellets out of the bag and setting them out in the air. Preferably to speed up the test, the pellets 25 are put under a conventional desk fan. The pellets are then subjected to practical observations to determine if there is excessive powdering and to determine if there is excessive If there is no powdering or wetness, the viscosity of the oil can be considered to be above the minimum If the surface is powdered or if the oil wicks 30 out from the material and the surface is wet, the viscosity is too thin, indicating that a more viscous carrier fluid should be added. In general, to obtain a suitable product,

different trials may be performed with carrier fluids having different viscosities depending on the degree of degradation observed.

Generally, the evaluation for physical properties can be made after the elapse of four hours. Degradation is easiest to observe around rough edges of the pellet. If the material shows no visible signs of degradation within four hours under a fan, the fragrant oil-carrier fluid will generally produce materials that have satisfactory physical properties, but if a higher degree of assurance is desired, a longer time may be employed for the observation.

10

For determining the fragrancing ability of the fragrant oil and carrier, the pellets are placed in a 10 ft. by 10 ft. (3.05 m by 3.05 m) room for about a day and a panel of experienced testers, selected for their acuity of smell, are asked for their impressions. If the room is judged by them to have a pleasant degree of fragrance by the end of the day, the fragrancing properties will be satisfactory.

20 If there are negative observations of physical properties or fragrancing capabilities, change in the carrier fluid of the fragrant oil is made to provide more fragrancing or better physical properties.

In general, a higher viscosity carrier fluid is used if it is desired to enhance the physical properties, decrease fragrancing power, and extend fragrancing duration, while a lower viscosity carrier is used to enhance the strength of smell by achieving a higher evaporative rate.

The amount of change of viscosity that is

appropriate depends on the type of fragrance and the carrier provided by the fragrance vendor. Four general categories of fragrance are often recognized: fruit, floral, spice and citrus. Citrus and spice oils usually are thinner than

floral and fruit oils and generally require addition of a more viscous carrier. The more viscous carriers include, for example, polysorbate (which is available in a variety of viscosities, a preferred grade used herein being Tween-20) or Hercolyn D. Less viscous carriers include, for example, dipropylene glycol or diethylphtlalate.

5

30

Carrier fluids that are known by the industry to work well (provide pleasant fragrancing in the liquid state) with a particular fragrance may not perform well on salt granules. For instance, with cinnamon fragrance, dipropylene glycol is often used in the industry.

Observations as described have indicated this carrier fluid is too thin in the particular circumstances of example 3. Addition of 7% Hercylon D, (as described in Example 3) produces a more viscous carrier that achieves better results.

Additional Uses

Pellets or loose granules may be glued together or onto other products; examples include: decorative wreaths,

20 baskets, wooden crafts, and other craft type items. The pellets may be glued together in combination to form shapes, such as a ball, teddy bear, tree, house, etc. Promotions like the scratch and sniff products are also possible. The pellets also can be pressed into desired shapes. A unique fragrance may be created by choosing a combination of differently treated pellets or granules and joining them together.

Uses for loose granules or powder include incorporation into potpourri, powdered chemical substances, roach powder or a rug cleaner, powdered pet deodorizers, and fragrancing scents. In addition, the powder may be used to coat surfaces (e.g. by electrostatic methods). Plastic garbage bags are one possible application. The powder may

- 20 -

also be incorporated within the plastic. Other uses include locker room balls or "Locker Balls" for YMCA's, health clubs, schools, and athletic teams (preferably with a strong citrus odor, e.g. lemon-orange) to mask the smell of sweat, mold, and body odor. Other uses include steam room or sauna 5 tablets, or "Spa Balls", for example, with menthol and/or eucalyptus oil. The Spa Balls™ would slowly melt, be about the size of golf balls (e.g. 400 grains) and would last three to five days. Other sizes and shapes for varied time uses could also be used, for example, a small 100 grain ball 10 that lasts just 1 day. Other uses include therapeutic uses, for example, aroma therapy, using a known stimulant type fragrant oil, such as lemon oil. The materials can also be employed as an insecticide or insect repellent by selection of certain herbal oils capable of insecticide or repellent 15 activity. For example, cedar oil can be used to repel moths, also pennyroyal, mint, wormwood, and lavender repel fleas and ticks, tansy repels ants. Southernwood oil can also be used as well as other insect repellant and 20 insecticide botanical oils.

What is claimed is:

- 21 -

1. A fragrant material for imparting a pleasant
2 fragrance when exposed in dry state at ambient conditions or
3 when placed in heated water, the fragrant material
4 comprising a molded shape-of compressed sodium chloride salt
5 granules having a fragrant oil dispersed within the molded
6 shape.

Š

- 2. The fragrant material of claim 1 comprising a plurality of said molded shapes in the form of pellets having an average size of approximately 1/4 inch (0.635 cm) to 1 inch (2.54 cm).
- The fragrant material of claim 1 wherein the granules of the molded shape comprises vacuum granulated sodium chloride.
- 4. The fragrant material of claim 1 additionally
 comprising a dye dispersed within the molded shape.
- 5. The fragrant material of claim 1, 2, 3 or 4 produced by the process of applying the fragrant oil to the surface of a pre-formed shape of compressed salt granules and allowing the oil to be absorbed into the pores between the granules.
- 6. The fragrant material of claim 5 wherein the process of production includes providing a plurality of said preformed shapes in the form of pellets and applying the fragrant oil thereto by spraying.
- 7. The fragrant material of claim 1, 2, 3 or 4 formed by the process of applying the fragrant oil to a

- 22 -

- 3 loose aggregation of granules of sodium chloride and
- 4 thereafter forming the granules into said molded shape.
- 1 8. The fragrant material of claim 7 formed by the
- 2 process of compressing the granules under high pressure to
- 3 form the molded shape.
- 9. A fragrant material for imparting a pleasant
- 2 fragrance when exposed in dry state at ambient conditions or
- 3 when placed in heated water, the fragrant material
- 4 comprising an aggregation of small vacuum granulated sodium
- 5 chloride granules and a fragrant oil dispersed within said
- 6 aggregation.
- 1 10. The fragrant material of claim 1 or 9 wherein
- 2 most of said sodium chloride granules are about 850 microns
- 3 or less in size.
- 1 11. The fragrant material of claim 1 or 9 including
- 2 a minor amount of finely divided, inert, water insoluble,
- 3 hygroscopic particles.
- 1 12. The fragrant material of claim 11 wherein said
- 2 finely divided inert particles are selected from the group
- 3 consisting of silicon dioxide, calcium silicate, magnesium
- 4 carbonate, tricalcium phosphate and sodium silicoaluminate
- 5 and mixtures and combinations thereof.
- 1 13. The fragrant material of claim 10 wherein said
- 2 finely divided inert particles have a particle size of about

3

3 10 microns.

PCT/US89/04958

- 1 14. The fragrant material of claim 11 wherein the
- 2 weight ratio of said finely divided particles to said
- 3 fragrant oil is about 1 to 3.
- 1 15. The fragrant material of claim 11 herein said
- 2 finely divided inert particles comprise about 2% or more by
- 3 weight of said fragrant material.
- 1 16. The fragrant material of claim 11 wherein said
- 2 finely divided inert particles comprise more than about 3%
- 3 and less than about 10% by weight of said fragrant material.
- 1 17. The fragrant material of claim 1 or 9 wherein
- 2 said fragrant oil includes an organic carrier fluid selected
- 3 from diethylphthalate, methyl dihydroabietate, Hercolyn D,
- 4 propylene glycol, dipropylene glycol, isoparaffins, Lozal,
- 5 saltrol, polysorbate, D-limonene, glycerin and Isobar M and
- 6 mixtures and combinations thereof.
- 1 18. The fragrant material of claim 17 wherein the
- 2 fragrant oil has a viscosity under ambient conditions on the
- 3 order of about 10 cps or more.
- 1 19. The fragrant material of claim 18 wherein said
- 2 viscosity is on the order of about 10 to 20 centipoise.
- 1 20. The fragrant material of claim 1 or 9 further
- 2 including a dye which is soluble in water.
- 1 21. The fragrant material of claim 20 wherein said
- 2 dye is soluble in oil.

- 24 -

Ē

1 22. The fragrant material of claim 21 wherein said 2 dye is a polymer-bound azo colorant.

- 1 23. The fragrant material of claim 1 or 9 wherein
- 2 said fragrant oil is selected to perform as a room
- 3 freshener.
- 1 24. The fragrant material of claim 1 or 9 wherein
- 2 said fragrant oil is selected to mask a pervading odor.
- 25. The fragrant material of claim 24 wherein said 2 odor is an animal odor.
- 1 26. The fragrant material of claim 1 or 9 adhered
- 2 as a coating to an object to which it is desired to impart a
- 3 fragrance.
- 1 27. The fragrant material of claim 1 or 9 wherein
- 2 said fragrance acts on an insect repellant.
- 1 28. The fragrant material of claim 1 or 9 wherein
- 2 said material is suitable for therapeutic use.
- 1 29. The fragrant material of claim 1 or 9 wherein
- 2 said material is a locker room ball.
- 1 30. The fragrant material of claim 1 or 9 wherein
- 2 said material is a steam room or sauna ball.
- 31. A dry, loose mixture comprising at least a
- 2 large number of small vacuum granulated sodium chloride
- granules, a relatively minor amount of finely divided,

- 25 -

- 4 inert, water insoluble, hygroscopic particles and a fragrant
- 5 oil dispersed in the mixture of said granules and particles.
- 1 32. The dry, loose mixture of claim 31 in the form
- 2 of rug freshener.
- 1 33. A fragrant material for imparting a pleasant
- 2 fragrance when exposed in dry state at ambient conditions or
- 3 when placed in heated water, the fragrant material
- 4 comprising an aggregation of small anhydrous salt granules
- 5 and a minor amount of finely divided, inert, water soluble,
- 6 hygroscopic particles, said finely divided inert particles
- 7 selected from the group consisting of silicon dioxide,
- 8 calcium silicate, magnesium carbonate, tricalcium phosphate
- 9 and sodium silicoaluminate and mixtures and combinations
- 10 thereof, said aggregation carrying a fragrant oil, the
- 11 weight ratio of said finely divided particles to said
- 12 fragrant oil being about 1 to 3, the salt granules in said
- 13 fragrant materials being selected so that in the presence of
- 14 said inert particles they are dimensionally stable under
- 15 ambient conditions.
 - 1 34. The fragrant material of claim 33 wherein said
- 2 aggregation is in the form of a compressed molded shape.
- 1 35. The fragrant material of claim 33 wherein said
- 2 aggregation is a loose mixture.
- 36. A method for forming a substantially dry
- 2 fragrant material comprising providing an aggregation of
- 3 small vacuum granulated sodium chloride granules and
- 4 applying to said aggregation a fragrant oil.

- 26 -

œ.

- 1 37. The method of claim 34 further including fusing
- 2 said granules into a molded shape by application of
- 3 pressure.
- 1 38. The method of claim 35 wherein said fragrant
- 2 oil is applied before fusing the granules.
- 1 39. The method of claim 37 wherein said fragrant
- 2 oil is applied after fusing said granules.
- 1 40. The method of claim 36 wherein said fragrant
- 2 oil is applied to loose granules and said material is
- 3 retained in a loose state.
- 1 41. The method of claim 36 further including
- 2 providing in mixture with said salt granules, a minor
- quantity of inert, water insoluble, hygroscopic particles
- 4 capable of increasing the amount of oil held in said
- 5 material while maintaining said material in a dry to the
- 6 touch state, said particles selected from the group
- 7 consisting of silicon dioxide, calcium silicate, magnesium
- 8 carbonate, tricalcium phosphate and sodium silicoaluminate
- 9 and mixtures and combinations thereof.
- 1 42. The method of claim 36 further comprising
- 2 formulating said fragrant oil to include a carrier fluid,
- 3 the fragrant oil having a resultant viscosity on the order
- 4 of about 10 cps or more.

International Application No. PCT US89/04958

I. CLASSIFICATION OF	SUBJECT	MATTER (if s	several classification	symbols apply,	indicate all) 6

According to Internations' Patent Classification (IPC) or to both National Classification and IPC Int. $C1.(5)\,A61K$ 7/46

II. FIELDS SEARCHED

Minimum Documentation Searched 7

1	William Botomentation General				
Classification System		Classification Symbols			
	512/3,4		-,		
US	424/76.3, 76.4				

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched 8

III. DOCUMENTS CONSIDERED TO BE RELEVANT 9

Category *	Citation of Document, 11 with indication, where appropriate, of the relevant passages 12	Relevant to Claim No. 13
х	US, A 3,371,984(Kelly) 5 March 1968	1-42
Α	US, A 3,943,243(Kook) 9 March 1976	1-42
Α	US, A 4,285,905(Feit) 25 August 1981	1-42
Α	US, A 4,071,616(Bloch) 31 January 1978	1-42
X	JP, A 58-112,541(Enklar) 5 July 1983	1-42
X	JP, A 60-99,256(Tsukamoto) 3 June 1985	1-42
Α	JP, A 58-185,161(Enklar) 28 October 1983	1-42
•		
:		
Ī		

- * Special categories of cited documents: 10
- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed
- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person seried in the art.
- "&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search

Date of Mailing of this International Search Report

31 JANUARY 1990 0 6 APR 1990

International Searching Authority

ISA/US

Signature of Authorized Officer

James Hleamer